CRACK INITIATION IN A NI-BASE SUPERALLOY

INTVELD, AJH; DEHOSSON, JTM; Huis in't Veld, A.J.

Published in:
Scripta Metallurgica

DOI:
10.1016/0036-9748(87)90287-0

Publication date:
1987

Scripta METALLURGICA Vol. 21, pp. 1481-1486, 1987 Pergamon Journals, Ltd.

CRACK INITIATION IN A Ni-BASE SUPERALLOY

A. J. Huis in't Veld, J. Th. M. De Hosson

Department of Applied Physics, Materials Science Centre,
University of Groningen,
Nijenborgh 18, 9747 AG Groningen, The Netherlands

(Received July 13, 1987)
(Revised August 28, 1987)

Introduction

Oxide dispersion strengthening (ODS) has been successfully combined with γ' precipitation hardening in the superalloy MA 6000. The development of MA 6000 is strongly connected with new processing techniques. The directional solidification process is used to produce columnar grains whereas ODS is a product of powder technology. Although undesirable impurities are removed by vacuum melting some irregularities are found in the alloy as received. Together with grain boundaries and the specimen outer surface these irregularities are sites of crack initiation during creep or fatigue testing.

During directional solidification impurities cause strings of small recrystallized grains. The effect of these strings lying in between large columnar grains will be analyzed. Moreover, spherical inclusions are found at fracture surfaces. It seems likely that these inclusions are formed during mechanically alloying followed by compaction of the raw materials.

In order to achieve high strength, barriers to dislocation motion are generated, e.g. γ' interfaces in Ni-base superalloys. On the other hand, each structural inhomogeneity causes stress concentration. Therefore, the optimum microstructure for high strength and fatigue resistance possesses a high degree of regularity thereby promoting homogeneous slip. Initiation of cracks may occur at slip band intrusions and extrusions which are formed if a persistent slip band reaches a free surface. Once a crack has been nucleated, the condition of brittle extension is governed by Griffith's theory [1] which balances the release of elastic energy against the work of fracture i.e. the surface energy of the new crack. Taking into account also the plastic work required to extend the crack makes Griffith's theory more compatible with fracture in metals.

Material

The chemical composition of the recently developed mechanically alloyed ODS superalloy MA 6000 is given in table 1 [2]. The MA 6000 material investigated is manufactured by Wiggin Alloys Ltd., England.

Table 1

Chemical composition of MA 6000 in weight%

Ni	Cr	Al	W	Ti	Mo	Y_2O_3	Zr	C	B
69	15	4.5	4.0	2.5	2.0	1.1	0.15	0.05	0.01

0036-9748/87 $3.00 + .00

Elemental analysis using energy and wavelength dispersive X-ray techniques proved small amounts of Re and Cu to be present. The alloy contains 2.5 vol% oxide particles (mean diameter 28 nm) and 50 to 55 vol.% γ' precipitates [3] with edge lengths of 200-300 nm (fig.1). In some specimens smaller γ' precipitates (25 nm) have been found.

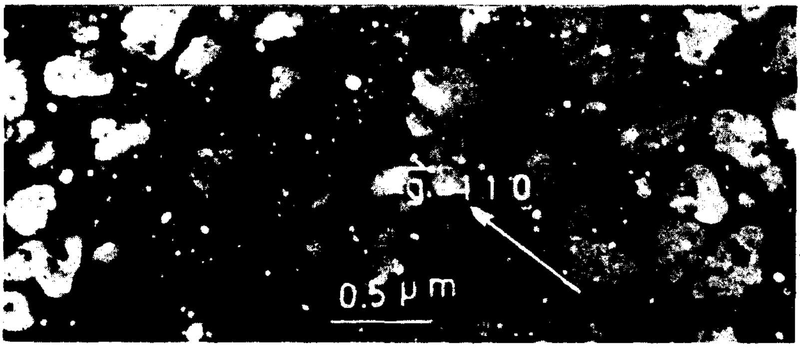

Fig. 1 γ' precipitates and oxide particles (small spheres) in MA6000.

The mechanically alloyed powders are compacted by hipping or extrusion. The as-consolidated material contains fine equiaxial grains that cause superplasticity with a maximum elongation of over 300% [4]. In order to develop optimum properties a large elongated grain structure is achieved by recrystallization. The recrystallized grains (fig. 2) are elongated with the large grain axis parallel to the working direction [5][6].

Fig. 2 Columnar grain structure of MA 6000 (left), recrystallized fine grains (middle), and imprints of impurities in the fine grains (right).

The columnar grains have a length of about 10 mm and share a <110> axis ± 20° [7]. The transverse grain size varies between 0.1 and 1 mm. Because of imperfect recrystallization strings of fine grains are formed (fig. 2b). Imprints of impurities in the fine grains are depicted in fig. 2c. γ' precipitates occupy the high angle grain boundaries of the fine grains. Within the columnar grains small angle (5°) boundaries have been identified with X-ray diffraction and transmission electron microscopy. X-ray diffraction on powder, chemically extracted, gives evidence for the presence of yttrium aluminium oxide ($YAlO_3$) and titanium tungsten carbide (Ti,W)C. These second phase particles may retard grain growth and stabilize the elongated grain structure. The final stage in manufacturing MA 6000 is a three step heat treatment consisting of 0.5 h at 1230 °C, AC (air-cooled), 2 h at 995 °C and 24 h at 845 °C, AC. This treatment develops the most favourable γ' structure.

Fracture surfaces investigated with SEM reveal spherical inclusions ranging in size from 1 to 20 μm. A few inclusions can be found on fracture surfaces (cross sectional area 12 mm^2) after fatigue at intermediate temperatures. However, sometimes they are grouped, as shown in figure 3. Because of the presence of such clusters of inclusions the mechanical strength turned out to be considerably lowered.

The elemental composition of most inclusions is not different from the overall alloy composition suggesting that they are formed during the process of mechanically alloying. Occasionally copper-rich inclusions have been found, the origin of which is not yet known.

Fig. 3 Inclusions on a fracture surface of MA 6000.

Crack Initiation During Creep Testing

Creep test specimens are prepared with the gauge length parallel to the longitudinal grain direction. The rectangular cross sectional area is about 1 mm^2; hence, a cross-section contains a limited number of grains which causes scatter in creep properties. Cavities form continuously throughout the creep process. However, in tertiary creep nucleation and growth of voids occurs at an accelerated rate. In MA 6000 cavities have been observed after creep at 538 MPa and 790 °C (see fig. 4). Nucleation of a cavity requires large stress concentrations which can be produced at the intersection of slip bands and grain boundary particles, in sliding grain boundaries and at grain boundary triple points [8]. It is obvious that the recrystallized fine grains associated with strings of impurities offer perfect nucleation sites for cavities, especially in the case of planar slip. The slip lines in figure 4a are parallel to the deformation induced twins which are formed by partial dislocations. Initially stacking faults are formed inside γ' precipitates [9], and as deformation goes on microtwins are formed.

Fig. 4 After creep the surface of a specimen shows slip lines, surface cracks and cavities which are associated with small grains. The cavities in fig. 4a (left) are magnified in fig. 4b (right).

A schematic representation of cavity formation is depicted in figure 5. SEM observations revealed that the grain boundaries of fine grains consist of agglomerated γ' particles. Tekin and Martin [10] have found that in HCF (high cycle fatigue) cracks grow along these boundaries by shearing γ' precipitates.

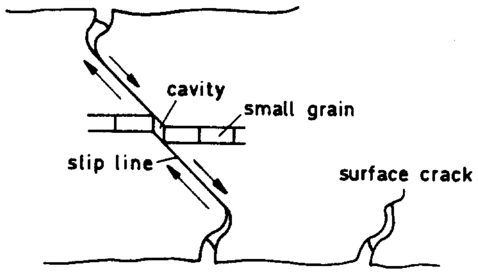

Fig. 5 Schematic representation of cavity formation.

Crack Initiation During HTLCF

High temperature low cycle fatigue experiments on MA 6000 have been performed in air with strain rate $\dot{\varepsilon}$ = $5.10^{-5}s^{-1}$. Strain controlled testing has been done on specimens with a gauge length of 18.0 mm and a diameter of 4.00 ± 0.05 mm. The surface finish is obtained by grinding, R_a ≈ 0.5 µm. The complete LCF (low cycle fatigue) data are published elsewhere [11]; here we will concentrate on the initiation of cracks.

Generally the irregularity of fracture surfaces appears to increase with increasing deformation temperature. At 760 °C and 850 °C the fracture surfaces are perpendicular to the applied stress whereas at 950 °C the angle between the direction of applied stress and the fracture surface is about 45°. At 1050 °C the highly irregular fracture surface is again perpendicular to the applied stress.

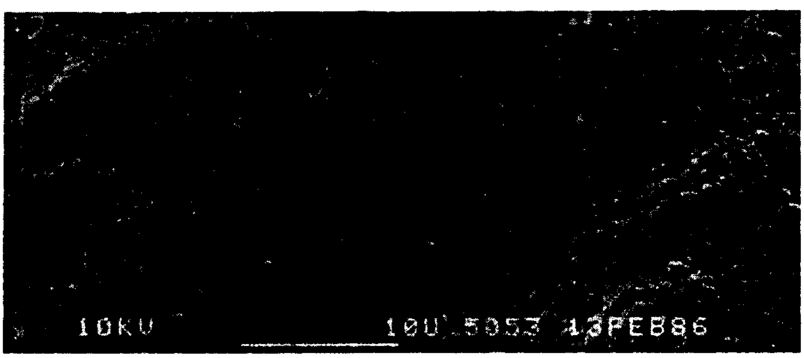

Fig. 6 Collinear particles on the stage II fracture surface after LCF at 850 °C, $\dot{\varepsilon}$ = $5.10^{-5}s^{-1}$, ε_{sw} = 0,40%, $\Delta\sigma/2$=450 MPa.

The site of fracture initiation can be found easily because steps in a fracture surface start at the point of initiation and appear as white lines in a SEM micrograph. In the high stress regime 465 MPa < $\Delta\sigma/2$ < 685 MPa at 760 °C fracture initiates at the surface of a specimen whereas in the low stress regime at 400 and 530 MPa initiation in the bulk has been observed. After initiation circular propagation causes a crater-like hole in the fracture surface. Outside the crater striations are observed, indicating stage II crack propagation.

On the stage II fracture surface fine powder is found. It is likely that this fine powder is formed by grinding the fracture surface, in agreement with the model for the formation of sawtooth striations by peak-peak contact during unloading [13]. This process may account for the rows of particles found sometimes on fracture surfaces (see fig. 6).

At 850 °C fracture initiates in the bulk for $\Delta\sigma/2$ = 370 and 388 MPa whereas at a higher stress (450 MPa) simultaneous initiation at the surface and in the bulk has been found. At 950 °C fracture initiates at the surface and is transgranular across about half the cross-sectional area. In the remaining half we have found deep cracks along grain boundaries. So in the second half fracture occurs along grain boundaries and by transgranular crack growth across the grain cross-section. At 1050 °C the whole fracture surface is covered with deep cracks along grain boundaries. Spherical inclusions like the ones depicted in fig. 3 have been found in the fracture surface. These inclusions seem to induce stress concentration. However, crack initiation has not been observed.

Discussion

Fatigue crack propagation is usually transgranular at low temperature, typically below 0.5 T_m. The transition to intergranular crack propagation is driven by grain boundary cavitation. Fatigue induced cavitation begins immediately after the start of cycling, without an incubation period. During part of a stress-strain loop creep processes will dominate. However, the damage mechanisms of creep and fatigue are not simply additive. Creep cavitation may be enhanced in the plastic zone of a fatigue crack whereas fatigue cracks may be nucleated at cavitated boundaries.

The site of crack initiation in the bulk during LCF at 850 °C (see fig. 6) has been investigated extensively. The "particle" which causes initiation is depicted in figure 7a. At first sight it seems reasonable to follow Hoffelner and Singer [2] and assume this "particle" to consist of matrix material. Afterwards, the fracture surface has been etched slightly and the same "particle" is depicted in figure 7b. From a stereo electron micrograph we have concluded that the "particle" consists of the usual bulk material whereas the outer surface consists of agglomerated γ' precipitates. Part of the outer surface is still visible in fig. 7b. Moreover, grain boundaries start at the particle, e.g. at the lower left hand side of the "particle" in figure 7b. It turns out that the "particle" is a small grain.

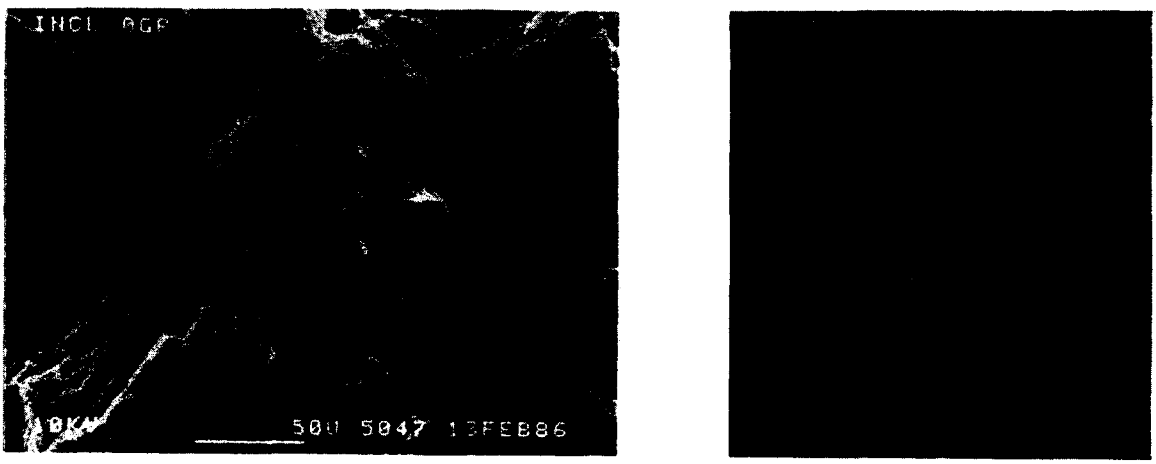

Fig. 7 A small grain which initiates fracture during LCF at 850 °C (a, left), the same grain slightly etched, part of the outer surface still visible (b, right).

Knowing that the "particles" which initiate fracture in the low stress regime at 760 °C and 850 °C are crystallized grains, we may conclude that these grains are limiting the fatigue properties of MA6000 at $\dot{\varepsilon} = 5.10^{-5}s^{-1}$. It is likely that cavitation at the grains, as observed after creep (fig. 4), causes fatigue crack initiation.

In the high stress regime at 760 °C and 850 °C slip bands cause initiation of fracture at the surface. At 950 °C and 1050 °C damage occurs at all grain boundaries and the effect of small recrystallized grains is overruled by pull out of the elongated grains. In conclusion: the small recrystallized grains are detrimental for fatigue properties at $\dot{\varepsilon}$-T conditions where intergranular initiation is not yet operative and where the stress is too low to cause surface initiation. In order to improve the fatigue and creep properties the growth of small recrystallized grains should be avoided. A homogeneous columnar grain structure may be obtained during directional solidification in the absence of impurities (oxides or carbides). Therefore, a reduction of the impurity level is of crucial importance.

Conclusions

In MA6000 fatigue cracks are nucleated at cavitated boundaries of small recrystallized grains during LCF at low stresses at 760 °C and 850 °C ($\dot{\varepsilon} = 5.10^{-5}s^{-1}$). The small grains are detrimental for fatigue properties at $\dot{\varepsilon}$-T conditions where initiation at columnar-grain boundaries is not yet operative and where stress is too low for surface initiation. In the high stress regime at 760 °C and 850 °C fracture initiates at the surface. At 950 °C and 1050 °C damage occurs at all grain boundaries. Spherical inclusions, like the ones in figure 3, have not been observed to initiate fracture.

In order to reduce the number of small recrystallized grains a reduction of the impurity level during directional solidification is of crucial importance.

Acknowledgements

The work is part of the research program of the Foundation for Fundamental Research on Matter (F.O.M.-Utrecht) and has been made possible by financial support from the Netherlands Organization for the Advancement of Pure Research (Z.W.O.-The Hague). Thanks are due to Mr. H. J. Bron for technical assistance.

References

1. A. A. Griffith, Phil. Trans. Roy. Soc. London 221A, 163 (1920).
2. Inco MAP, specification according to the manufacturer.
3. Y. G. Kim, H. F. Merrick, NASA CR-159493, May 1979.
4. J. K. Gregory, J. C. Gibeling, W. D. Nix, Met. Trans. 16A, 777 (1985).
5. R. F. Singer, G. H. Gessinger, Met. Trans. 13A, 1463 (1982).
6. R. K. Hotzler, T. K. Glasgow, Met. Trans. 13A, 1665 (1982)
7. T. E. Howson, D. A. Mervyn, J. K. Tien, Met. Trans. 11A, 1609 (1980).
8. E. H. v. d. Molen, J. M. Oblak, O. H. Krieger, Met. Trans. 2, 1627 (1971).
9. A. J. Huis in't Veld, P. M. Bronsveld, G. Boom, J. Th. M. De Hosson, ICSMA 7, Pergamon Press, 1985, p. 411.
10. A. Tekin, J. W. Martin, Conf. High Temperature Alloys for Gas Turbines and Other Applications, Liège, 1986.
11. A. J. Huis in't Veld, Thesis, Un. Groningen, 1987.
12. W. Hoffelner, R. F. Singer, Met. Trans. 16A, 393 (1985).
13. H. Mughrabi, R. Prass, H.-J. Christ, D. Puppel, Atomistics of Fracture, Bad-Reichenhall, 1986.